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# Advances in Thin Film Thermocouple Durability Under High Temperature and Pressure Testing Conditions

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# ADVANCES IN THIN FILM THERMOCOUPLE DURABILITY UNDER HIGH TEMPERATURE AND PRESSURE TESTING CONDITIONS

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## SUMMARY

Thin film thermocouples for measuring material surface temperature have been previously demonstrated on several material systems and in various hostile test environments. A well-developed thin film fabrication procedure utilizing shadow masking for patterning the sensors elements had produced thin films with sufficient durability for applications in high temperature and pressure environments that exist in air-breathing and hydrogen-fueled burner rig and engine test facilities. However, while shadow masking had been a reliable method for specimens with flat and gently curved surfaces, it had not been consistently reliable for use on test components with sharp contours. This work reports on the feasibility of utilizing photolithography processing for patterning thin film thermocouples. Because this patterning process required changes in the thin film deposition process from that developed for shadow masking, the effect of these changes on thin film adherence during burner rig testing was evaluated. In addition to the results of changing the patterning method, the effects on thin film adherence of other processes used in the thin film fabrication procedure is also presented.

## INTRODUCTION

The continuous development and evaluation of both space and aeronautics propulsion systems requires surface temperature measurement techniques that are reliable and durable in the hostile test conditions typically encountered in burner rig and test engine environments. Reliable surface temperature data enables experimental verification of analytical techniques that have been created to determine surface temperature distribution. Such information is relevant to several research areas, including materials and structures evaluation, and fluid mechanics.

Ideally, a surface temperature measurement device that is used in burner rig or engine tests should not disturb the conditions at the surface during testing. Wire sensors mounted on the test surface may significantly disrupt the flow conditions. This can be overcome by installing the sensors in machined grooves in the test structure; however, the heat transfer and temperature distribution profiles may be compromised. In addition, this procedure can alter the structural integrity of the test component which is often unacceptable in burner rig and test engine situations.

Thin film thermocouples are intended to minimize the negative effects of placing a sensor on the surface of a test structure. These sensors do not require that the surface be machined and are many orders of magnitude thinner than wire sensors. As a result, thin film thermocouples add negligible mass to the surface and create minimal disturbance of the gas flow over the surface.

At NASA Lewis Research Center, thin film thermocouples were initially developed for superalloy turbine blade applications for air-breathing propulsion systems (ref. 1). The technology has since been extended to other high temperature material systems, including ceramic, intermetallic and ceramic matrix composite systems (refs. 2 to 3). Additionally, the technology has been demonstrated in hydrogen-fueled test environments (refs. 2 and 4). The test configurations have ranged from testing sensors on coupons in static furnace conditions to sensors on flat specimens and complex shaped components in burner rig and engine test environments.

The sensors utilize platinum-13% rhodium (Pt-13% Rh) versus platinum (Pt) thermocouple elements (standard Type R) for high temperature capability and are sputter deposited directly onto the test specimen or component. Thin film thermocouples have traditionally been patterned using shadow masking techniques on test components with curved surfaces, including turbine blades and combustor liners. However, shadow masking with stenciled stainless steel shim stock has proven cumbersome and limiting when instrumenting a component containing sharp contours. It is difficult to clamp a stiff metal mask to the substrate so as to sufficiently maintain constant mask-to-surface contact across the entire pattern which is needed to prevent shadowing of the deposited metal film. Additionally, the heat present during sputter deposition can distort the mask leading to shadowing of the deposited film. If several thermocouple elements are stenciled in the metal mask, decreased mask stiffness results making it difficult to sufficiently clamp the mask between the elements to prevent distortion. Also, as finer line widths are needed for smaller thermocouples, the mask thickness must be decreased to prevent shadowing from the pattern edge line. Because thinner masks are less stiff, susceptibility to heat distortion increases. An example of a component with sharp contours is the turbine blade on which the shoulder separating the airfoil and shank areas protrudes out from the blade surface at a sharp angle and presents limited space for clamping a metal mask. Space shuttle main engine turbine blades have been shadow masked for thin film thermocouples with great difficulty over this blade area resulting in low success rates when fabricating working thin film sensors (refs. 2 and 4). It is preferable to have an alternative masking method that is reliable for use on these types of challenging shapes.

The work presented herein discusses the viability of using photolithography masking techniques to pattern thin film thermocouples for applications in burner rig and engine testing. Photolithography employs patterned photomasks which are placed on the surface after application of photoresist and are then exposed to ultraviolet light. The component with a patterned layer of photoresist is then deposited with the sensor material. Because exposure occurs at room temperature and the photomask is no longer needed after patterning the photoresist, mask distortion during sputter deposition is not a concern as it is for metal masks. Photomasks also offer flexibility in that patterns with fine line geometries are achievable. This is valuable in applications where several thermocouples are needed in a small space which dictates that the sensor element widths be smaller than what is reliably feasible with metal masks. Flexible photomasks have been fabricated with mylar film which conform to the component shape thereby making the photolithography process applicable to curved surfaces (ref. 5).

The fabrication procedure utilizing shadow masking that had been developed for the Pt-13% Rh versus Pt thin film sensors has produced films that were successfully tested in various test environments. However, this film deposition procedure had to be altered to accommodate the temperature limits imposed by the materials used in the photolithography process. The resultant films were then tested for durability on flat specimens in a hydrogen-fueled rocket engine test facility. The test facility was altered to provide high pressure conditions in a specimen test chamber. Previously reported tests in this facility limited the specimens to high temperature gas exposure while mounted in the engine exhaust plane (refs. 2 and 4). While this test was designed for a reusable launch vehicle application, the results are relevant to thin film sensor applications for air breathing propulsion components as well.

In addition to investigating the effect on film durability of altering the deposition parameters to accommodate the photolithography masking technique, two additional fabrication parameters were looked at to determine if there was any influence on film durability during high temperature and high pressure testing. The effect of using fine powder grit blasting to prepare the surface prior to film deposition was examined to determine if it aided film adhesion during testing. Additionally, two deposition methods, sputter deposition and electron beam evaporation, were compared to determine the effect, if any, of deposition method on the durability and adhesive strength of the deposited aluminum oxide layer. The function of this layer was to electrically insulate the thermocouple elements from the conducting substrate material.

## SENSOR FABRICATION

As shown in the schematic in figure 1, thin film thermocouples are composed of several layers. The substrates,  $2.5 \times 12.7 \times 0.318$  cm ( $1 \times 5 \times 0.125$  in.), were a nickel-based superalloy, Mar-M 246, which is stock material for first stage turbine blades used in the high pressure fuel turbopump of the space shuttle main engine. The metal thermocouple elements were electrically insulated from the metal substrate by a layer of aluminum oxide. Several steps were used to prepare this layer. A  $125 \mu\text{m}$  ( $0.005$  in.) thick coating of PWA 270 NiCoCrAlY (12 wt% Co, 18 wt% Cr, 12 wt% Al, 0.3 wt% Y and balance Ni) was electron beam vapor deposited onto the substrates. The coating was then glass bead peened to increase its density. Half of the twenty samples were grit blasted with  $27 \mu\text{m}$  aluminum

oxide powder in order to study the effects on film adhesion of lightly roughening the coating. All of the samples were then cleaned in microsoap and deionized water and heat treated in vacuum at 1000 °C (1800 °F) to facilitate migration of aluminum to the surface. This was followed by an oxidation heat treatment in air at 1000 °C (1800 °F) to thermally grow aluminum oxide with a thickness on the order of 1 to 3 μm. To form a pinhole-free, crack-free electrically insulating film, an additional layer of aluminum oxide was deposited onto the grown aluminum oxide.

For the remaining processing steps, a matrix of fabrication steps was created in order to determine the effects, if any, of each process on film adhesion properties during testing in harsh conditions (table I). For each set of samples, one processing parameter was changed while the other parameters were held constant. Sputter deposition was used to deposit aluminum oxide on half of the samples and electron beam vapor deposition was used for the remaining samples. Sputter deposition occurred in the diode mode in pure argon at a power density of 3.09 W/cm<sup>2</sup>. The sputtered aluminum oxide films were ~2 to 3 μm thick. For deposition by the electron beam vapor deposition process, a substrate heater was maintained at 900 °C. The evaporated films were ~4 to 5 μm thick. The electron beam vapor deposition process offers the advantage of being a much faster process than sputter deposition with process times of <1 hr versus 10 to 20 hr for depositing films of these thicknesses. The samples were then ready to be patterned for the thermocouple elements prior to metal deposition.

Per the matrix in table I, half of the samples were patterned with 0.025 cm (0.010 in.) thick stainless steel shim stock masks that had been machined with a thermocouple element pattern. The metal masks were clamped onto the sample surface and sputter deposited with a 3 to 5 μm thick platinum layer. The metals were sputter deposited in magnetron mode at a power density of 2.47 W/cm<sup>2</sup>. A heater located at an adjacent sputter station was maintained at 400 °C. For the first several thousand angstroms of deposited metal, oxygen-enhanced sputter deposition was used to aid film adhesion to the aluminum oxide layer. The masking and deposition process was then repeated for the platinum-13% rhodium element.

The remaining samples were patterned via the photolithography process. For the liftoff technique, positive photoresist was first applied to the surface. Because this patterning was for the deposition of relatively thick (3 μm) metal films, the photoresist layer was thicker (~3 μm) than that typically used in photolithography processes for the microelectronics manufacturing industry (<1 μm). Subsequent steps included exposure to ultraviolet light of the photoresist through a patterned mask and processing in developer to remove the exposed photoresist. Some undercutting occurred at the photoresist line edges that would later allow greater ease in removing the photoresist and overlying deposited metal. The specimens were then baked at low temperature (90 °C, 190 °F) to remove any remaining moisture from the photoresist layer.

The use of positive photoresist for patterning the thermocouple elements limits the temperature to which the sample can be exposed during sputter deposition. As a result, some deposition parameters were altered to prevent overheating and subsequent damage to the photoresist. When photoresist is damaged in this way, the solvent is unable to remove the photoresist and overlying metal film. During metal deposition, the specimens were positioned on a cooled substrate holder maintained at 0 °C. Platinum was sputter deposited in diode mode at a power density of 1.10 W/cm<sup>2</sup>. Oxygen-enhanced sputter deposition was used for the first several thousand angstroms of deposition and the metal films were ~3 to 4 μm thick. After deposition of the platinum layer, the photoresist and overlying metal were removed by soaking in acetone. The processing steps were then repeated for the platinum-13% rhodium layer.

Despite steps taken to prevent overheating the photoresist during deposition, the samples were not evenly cooled across the surface resulting in some areas of photoresist damage. Consequently, the solvent was unable to completely remove the photoresist from the entire surface. This was evident by scattered metal deposits on the surface as well as poor edge lines for some of the thermocouples. In severe cases, lines of scattered metal deposits crossed between the two thermocouple elements resulting in shorting between the elements.

Figure 2 is a photograph of a sample instrumented with a thin film thermocouple element pattern. The individual element length and width were 10.64 cm (4.188 in.) and 0.159 cm (0.063 in.), respectively. The junction at which the two elements met was the surface temperature measurement point and was ~0.318 cm (0.125 in.) by 0.635 cm (0.250 in.). The thermocouples were fabricated with enlarged leadwire pads to allow greater ease in lining up the bare leadwires with the thin film elements prior to clamping and installation into the test rig.

In all, there were eight groups of specimens, each representing a unique set of deposition parameters (table I). All of the specimens were annealed for 20 hr at 1000 °C (1800 °F). The leadwires were 75 μm (0.003 in.) diameter platinum-13% rhodium and platinum bare wires that were clamped to the films prior to installation in the rocket laboratory facility for testing.

## ROCKET LABORATORY TEST FACILITY

The rocket lab facility in which the specimens were tested utilizes a gaseous hydrogen and oxygen rocket combustor to generate high temperature, high heat flux, hydrogen- or oxygen-rich hot gas environments (ref. 6). This facility approximates the thermal shock conditions of the space shuttle main engine turbopumps. Gas temperatures ranging from about 1000 to 2700 °C (1800 to 5000 °F) are obtainable at combustion chamber pressures up to 4.1 MPa (600 psi). For these tests, the engine was operated at fuel-rich ratios ( $O_2:H_2$  mass flow rate ratio of 1.0) resulting in gas temperatures of ~900 to 1100 °C. Unlike previous tests in which the samples were mounted in the exhaust plane of the engine (refs. 2 and 4), the samples were mounted in specially designed hardware within the combustion chamber. This provided for testing the specimens under higher pressure conditions of ~3.4 MPa (500 psi). The facility was instrumented with a pressure sensor for monitoring the pressure profile within the combustion chamber. The thin film thermocouple junction was located approximately in the center of the gas flow within the combustion chamber. The test duration was 3 sec/run. The test runs were reduced to 3 sec from 5 sec used in previous tests (refs. 2 and 4) due to concerns with overheating of the test chamber hardware. If a film was intact after one 3-sec test run, it then underwent additional test runs.

For the initial tests, the samples were mounted with the sensor surface positioned parallel to the gas flow. Those samples that performed well under these conditions were then tested with the sample surface positioned at a 22.5° angle into the flow. Additional testing was performed on the surviving films with the sample surface at a 45° angle into the flow. This position allowed for greater impact of the combustion gasses on the thin film sensors. Not all of the prepared samples were tested in the facility. Those samples that did not give temperature output due to a break in film continuity were not tested.

## RESULTS AND DISCUSSION

The three fabrication processes outlined above were evaluated for effect on film durability under high temperature and high pressure conditions. Sputtered aluminum oxide films and electron beam vapor deposited aluminum oxide films did not demonstrate any appreciable difference in ability to aid the adhesion of the thermocouple elements to the substrate during testing. This evaluation was based upon comparing all of the samples that were processed with sputtered deposited aluminum oxide with those that had electron-beam vapor deposited aluminum oxide. Approximately the same percentage of each set of samples had intact films after testing. The samples that had been grit blasted prior to thin film processing demonstrated greater durability than those samples that had been left as is. The former set of samples had about 50 percent with intact films versus only 20 percent with intact films for the latter set. The light roughening of the NiCoCrAlY surface may have allowed for some degree of mechanical interlocking between the deposited aluminum oxide insulating layer and the underlying NiCoCrAlY and thermally grown aluminum oxide layers.

Several samples patterned with the photolithography process performed very well during testing. A set of three samples which had processing procedures that included grit blasting the NiCoCrAlY coating, sputter deposition of aluminum oxide, and the photolithography mask survived all of the test runs in the rocket lab process (sample group no. 4 in table I). A total of six test runs each were conducted on two of the samples and the third underwent three test runs. The sample positions varied between test runs and included orientations of 0, 22.5, and 45° angles to the gas flow. All of the films were intact at the conclusion of the tests. While the focus of these tests was film adhesion and durability, temperature data were also collected. Figure 3 includes surface temperature and pressure data measured in the combustion chamber during one test run. The sample was oriented at a 0° angle to the gas flow. The thin film thermocouple measured a maximum surface temperature of approximately 580 °C (1080 °F) prior to shutdown. The nominal chamber pressure was ~3.5 MPa (510 psi). Because the test run was for 3 sec only, it is expected that the surface temperature would not approach the gas temperature, therefore the measured surface temperature is reasonable. The apparent temperature spike in the data after shutdown was present in the data from all of the test runs and is likely due to some type of electrical interference in the instrumentation system. With subsequent test runs, the maximum surface temperature output did demonstrate gradual degradation, even as the samples were oriented towards the flow. This may be attributable to gradual erosion of the thin film material during each additional test run thus affecting the sensor accuracy. This effect can be delayed with the application of a protective aluminum oxide overcoat film that has been utilized in previous work (refs. 2 and 4).

Because traditional wire thermocouples have proven unable to withstand the test conditions in this facility, no gas temperature data could be collected simultaneously to verify the thin film thermocouple data. However, the fact that the temperature profile in figure 3 corresponds to the chamber pressure profile indicates that the thin film thermocouples were operating normally. This marks the first time that temperatures within the combustion chamber could be measured in this facility.

While the films patterned with photolithography performed well in these tests, improvements are still needed to make this a reliable, repeatable process. In order to prevent potential shorting across the thermocouple elements due to stray metal deposits outside the elements pattern, the process for removing the photoresist after deposition must be improved. More consistent cooling across the specimen during sputter deposition is needed. Additionally, other solvents such as commercial strippers can be investigated that may remove heat-damaged photoresist more easily without causing damage to the deposited metal films. Finally, increasing the thickness of the photoresist layer relative to that of the metal film may also increase ease of photoresist removal.

Since the metal deposition parameters used for the specimens patterned with photolithography produced films durable for applications in burner rig and engine testing, it would be of interest to determine if these same parameters coupled with metal shadow masks would also provide durable films. Of particular interest is the effect of substrate cooling during sputter deposition on the film adherence since past work had demonstrated the effectiveness of substrate heating in forming highly adherent films. It is expected that substrate cooling would effectively eliminate the metal mask distortion that occurs with substrate heating and the higher power densities used for the shadow masked specimens. This should then reduce the occurrence of film shadowing outside of the patterned thermocouple elements. Additionally, thinner metal masks could then be used for multiple thermocouples and smaller line widths. If films fabricated with these deposition parameters proved to be consistently durable when used with either shadow masking or photolithography, this would allow the flexibility of utilizing either patterning method as needed for any particular application. Additional sensor fabrication and testing is required to determine the durability and reliability of such films.

## CONCLUSIONS

The preliminary work described herein demonstrated that replacing the shadow mask procedure with a photolithography mask procedure is a viable means for patterning thin film thermocouples to be used in high temperature, high pressure burner rig tests. Films fabricated with deposition procedures designed to accommodate the low temperature requirements for using photoresist proved to be durable in a harsh test environment. While a possible method for patterning thin film sensors on complex shaped components, improvements are needed to make photolithography a reliable, repeatable process. In particular, the process for removing the photoresist after deposition must be improved. The deposition process used for photolithography may also have potential for use with shadow masking. Additional work is needed to determine the viability of using the low temperature deposition process for both photolithography and shadow masking techniques. The effect of fine powder grit blasting in improving thin film adherence was also demonstrated. A comparison of deposition methods for aluminum oxide did not yield a clear advantage of either sputter deposition or electron beam vapor deposition in improving film adherence. Finally, these tests with thin film thermocouples marked the first time in which surface temperature was measured within the combustion chamber of this rocket lab test facility. Previous use of wire sensors had proven unreliable and insufficiently durable to withstand the harsh environmental conditions.

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TABLE I.—FABRICATION PROCESSES FOR TEST SAMPLES

Sample group number	Number of samples	Thermocouple patterning technique	Grit blast preparation	Al <sub>2</sub> O <sub>3</sub> deposition process
1	2	Shadow masking	None	Sputter deposition
2	3	Photolithography	None	Sputter deposition
3	2	Shadow masking	27 μm Al <sub>2</sub> O <sub>3</sub> powder	Sputter deposition
4	3	Photolithography	27 μm Al <sub>2</sub> O <sub>3</sub> powder	Sputter deposition
5	3	Shadow masking	None	E-beam vapor
6	2	Photolithography	None	E-beam vapor
7	2	Shadow masking	27 μm Al <sub>2</sub> O <sub>3</sub> powder	E-beam vapor
8	3	Photolithography	27 μm Al <sub>2</sub> O <sub>3</sub> powder	E-beam vapor

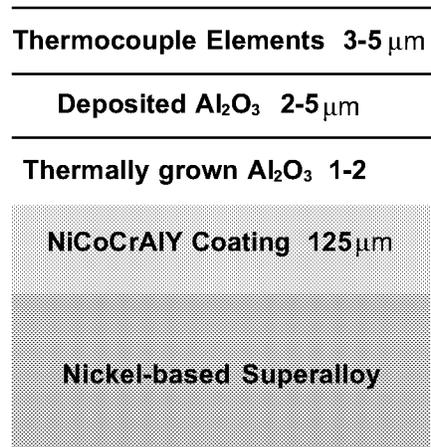


Figure 1.—Schematic of thin film thermocouple layers.

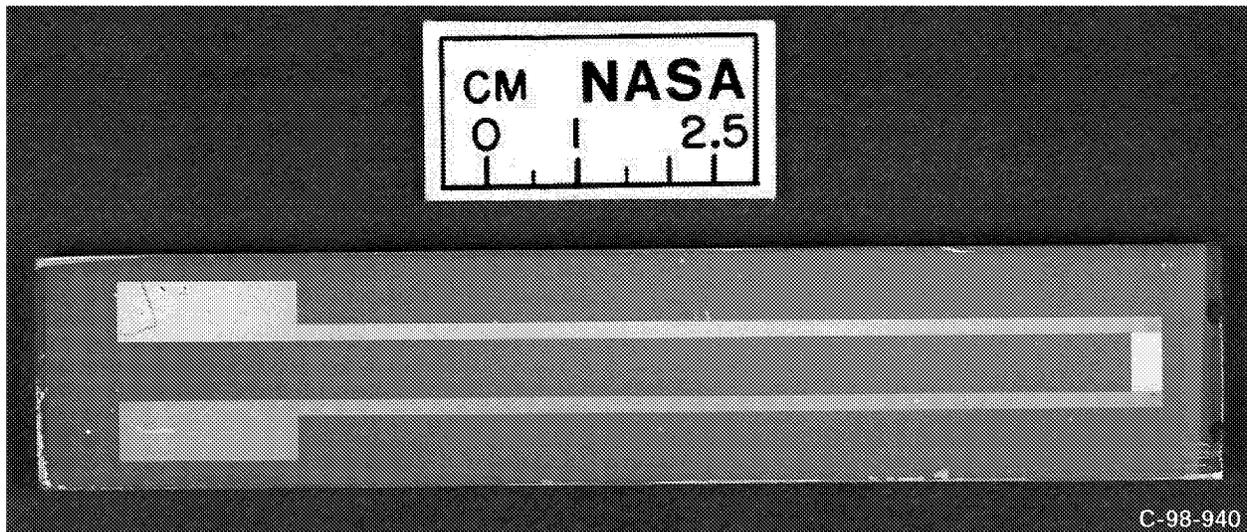


Figure 2.—Test sample instrumented with thin film thermocouple.

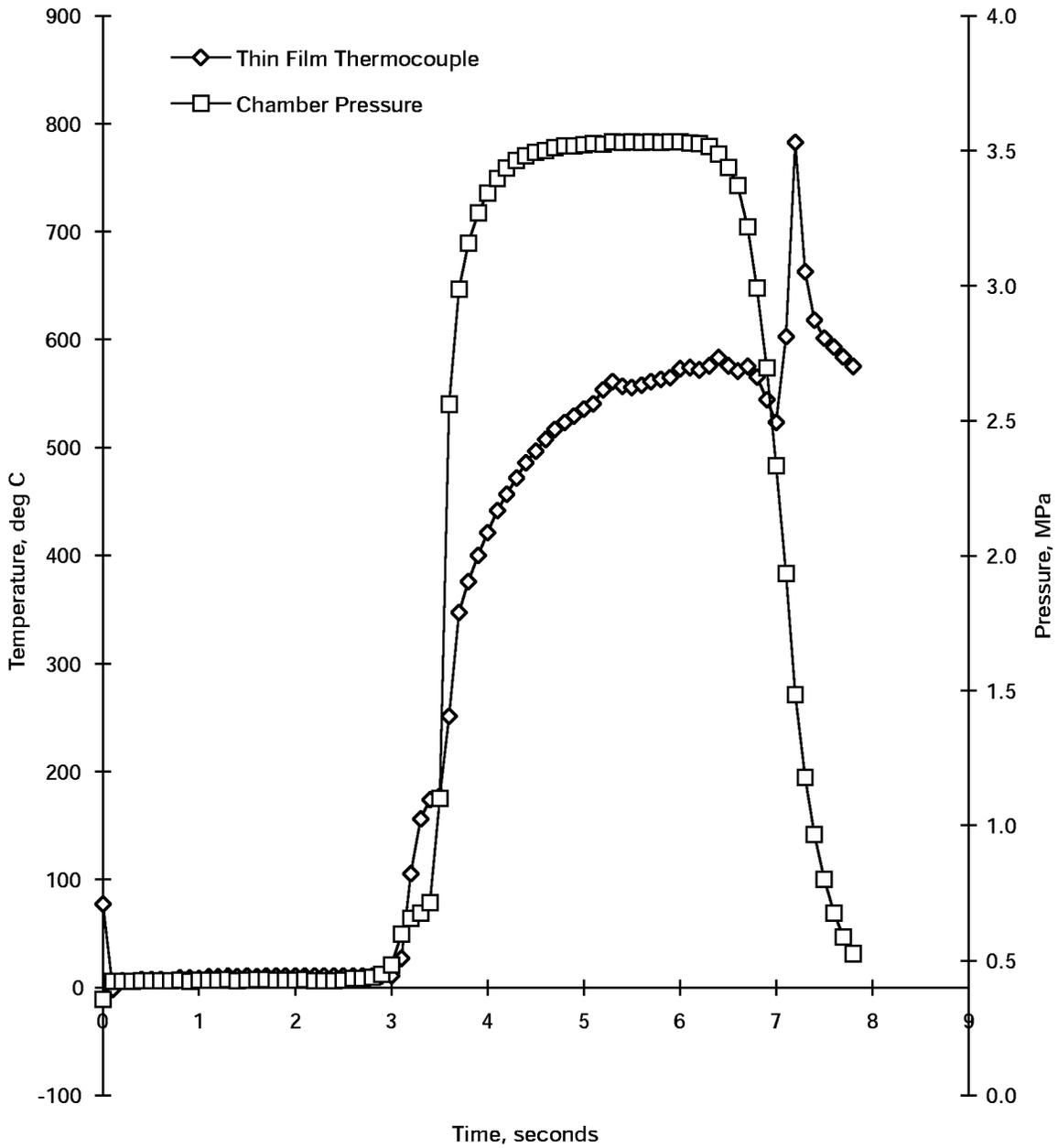


Figure 3.—Temperature and pressure data from thin film thermocouple burner rig test.

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